

## **Fat (Crude) or Ether Extract in Dried Milk Products**

### Scope

This method is used to determine the percentage of fat in dried milk products.

### Summary

The sample is reacted with ammonium hydroxide and then extracted with ethyl ether and petroleum ether. The corrected weight of fat is used to determine the percentage fat.

### Comments

A blank must be run with each set of samples. The difference between the duplicate determinations obtained simultaneously by the same analyst should be less than or equal to 0.2 g of fat per 100 g of sample. The sample must be ground.

### Apparatus and Materials

- A. Florence flask.
- B. Glass beads.
- C. Fat extraction flask or tube.
- D. Heated water bath, 60-70°C.
- E. Cooling bath.
- F. Steam bath.
- G. Oven,  $102 \pm 2^\circ\text{C}$ .
- H. Vacuum oven, 70-75°C.

Reagents

- A. Ammonium hydroxide, concentrated.
- B. Ethanol: 95%.
- C. Ethyl ether.
- D. Petroleum ether.
- E. Ethers mixture: a one to one mixture of ethyl ether and petroleum ether.

Procedure

- A. Weigh approximately 1.0 g of the sample (to the nearest 0.0001 g) into a fat extraction flask or tube.
- B. Add 8.5 ml of deionized water to the sample.
- C. Add 1.5 ml of concentrated ammonium hydroxide to the tube.
- D. Heat the tube and sample in a water bath for 15 minutes at 60-70°C. Shake the tube frequently.
- E. Cool the tube and sample to room temperature.
- F. Add 10 ml of ethanol to the sample and mix well.
- G. Add 25 ml of ethyl ether, stopper the tube and shake vigorously for one minute.
- H. Cool to room temperature if necessary.
- I. Add 25 ml of petroleum ether, stopper the tube and shake vigorously for one minute.
- J. Let the tube stand until the upper liquid is practically clear.
- K. Decant the ether layer into a 150 ml florence flask containing a few glass beads.

- L. Wash the lip of the tube and stopper with the ether mixture. Add the washings to the florence flask.
- M. Add 4 ml of ethanol to the sample.
- N. Repeat the extraction of the liquid remaining in the tube twice more. Use 15 ml of each solvent each time and add deionized water if necessary to bring the aqueous layer in the tube to original volume.
- O. Omit the rinsing with the mixed ethers after the final extraction.
- P. Evaporate the solution completely on a steam bath that does not cause spattering or bumping.
- Q. Dry the fat to a constant weight in an oven at  $102 \pm 2^{\circ}\text{C}$  or a vacuum oven at  $70\text{--}75^{\circ}\text{C}$  under a pressure of less than 50 mm Hg. Cool the flask in a desiccator.
- R. Weigh the cooled flask (to the nearest 0.0001 g) without wiping immediately before weighing.
- S. Remove the fat completely from the flask with 15-25 ml of warm petroleum ether.
- T. Dry the flask as in step Q and cool in a desiccator, and then weigh to the nearest 0.0001 g.

#### Calculations

- A. Determine the weight of the fat by subtracting the weight of the flask (step T) from the weight of flask and fat (step R).
- B. Determine the blank factor by subtracting the weight of the flask as found in (step T) from the weight of the flask plus blank as found in (step R).
- C. Calculate the corrected weight of fat by subtracting the blank (calculations step B) from the weight of the fat (calculations step A).
- D. The percentage of fat is the corrected weight of fat (calculations step C) divided by the weight of sample (procedure step A) and multiplied by 100.

Quality Control

- A. Check temperatures.
  - 1. Water bath should be 60 - 70°.
  - 2. Check drying oven before use with a calibrated thermometer and document on the worklist.
- B. Monitor time.
  - 1. Extraction tubes should be held in heated water bath for 15 minutes.
  - 2. Flasks and fat should be dried long enough to give a constant weight.

Bibliography

Official Methods of Analysis (1984) 14th Ed., AOAC, Washington, D.C., secs. 7.064